

## REMARKS

### I. Telephone Interview

The courtesy of Examiner Weier in discussing this application with the Applicants' representative in a telephone interview on February 27, 2008 is acknowledged with appreciation.

### II. Claim Amendments

Claims 1-5 and 18 are presently pending and previously withdrawn claims 6-17 are cancelled without prejudice. With this response, claims 1, 2, and 18 are amended. Support for these amendments are discussed below.

### III. 35 USC 112, second paragraph

Claims 2 and 18 are rejected under 35 USC 112, second paragraph for being indefinite.

The Examiner contends that in claim 2, the term "commonly used vegetable oils" is indefinite in that it is not clear what vegetable oils are "common". In reply the Applicants have amended claim 2 to recite "protecting ~~commonly used~~ vegetable oils selected from the group consisting of soybean oil, safflower oil, sunflower oil, and groundnut oil". Support for this amendment is found in withdrawn claim 16, which is now cancelled with this response.

The Examiner contends that in claim 18, it is not clear whether or not the recitations "such as alcohols. Esters, ketones" and "such as pure ethanol/ethylene glycol/propylene glycol" are actual claim limitations or not, and that it is also not clear whether the solvent and carrier, are any one of these compounds or whether it is considered a combination of all three. In reply, the Applicants have amended claim 18 to recite "...an organic solvent[[s]] selected from the group consisting of ~~such as~~ alcohols,[[.]] esters, ketones, substituted hydrocarbons and combinations thereof...", wherein said amendment clarifies that the organic solvent can be one of these or combinations thereof. Support for this amendment is found, *inter alia*, at page 7, lines 24-25. Claim 18 is further amended to recite "...a permitted carrier selected from the group consisting of ethanol, ethylene glycol, propylene glycol and combinations thereof...", wherein it is clear that each of the three listed carriers could be selected individually or in combination. Support for this feature as now amended is found, *inter alia*, at page 21, line 3 and page 24, lines 26-27 of the application. The Applicants have further amended claim 18 as follows "storing[[ed]] the dissolved extract under refrigeration until use" in order to further clarify the last step.

**IV. 35 USC 102(b) and 102(e)**

The Examiner contends that claims 1-5 and 18 are rejected under 35 USC 102(b) and (e) as being anticipated independently by Purdy et al. and JP 62-172-86. The Applicants respectfully traverse this rejection as follows.

With this response, the Applicants have amended claims 1 and 18 to recite “*consisting essentially of 5-20% lignan containing sesamol 10-16%; sesamin 60-75%; sesamol 5-8.8%...*”. At paragraph [009] of the present application, it is acknowledged that sesame extracts have been disclosed in the art, but that a sesame extract for effective preservation of food containing *sesamol 10-16%* has not been disclosed as in the present application. Thus, a product containing only the claimed components has not previously been disclosed. With the removal of the term “*essentially*”, additional ingredients are excluded by the proposed limitation (see MPEP 2111.03).

**Purdy et al.**

Purdy et al disclose a process for the extraction of sesame from sesame oil. The Examiner contends that it is expected that the sesame extract of Purdy et al would possess the same composition as in the presently claimed composition.

The Applicants disagree and submit that as amended herein, claim 1 and claim 18 in part recite, a composition “*...consisting of 5-20% lignan containing sesamol 10-16%; sesamin 60-75%; sesamol 5-8.8%, and balance being tocopherols, polyphenols/ferulic acid, denatured proteins, sugars, lipids, minerals and browning products*”.

Purdy et al. do not disclose a composition possessing lignan containing sesamol 10-16%. Purdy et al disclose a product that is “*primarily a mixture of sesamin and sesamol...*”. Since it was known at the time of filing that sesame oil contains “*sesamin, sesamol and sesamol*” (see col. 1, lines 28-31), Purdy et al had the capability of detecting sesamol, and a skilled artisan would certainly have reasoned that if Purdy intended to teach a product containing sesamol, he would have stated so. Applicants thus submit that Purdy in fact *teaches away* from including sesamol and that no skilled person would understand the product of Purdy et al to possess “*sesamol 10-16%*”, as recited in claims 1 and 18. Applicants thus respectfully disagree that the product of Purdy et al would “*inherently*” include “*sesamol 10-16%*” and submit that claims 1 and 18 are therefore not in fact anticipated by Purdy et al, and that at least by virtue of their dependency on claim 1, claims 2-5 are also not anticipated by Purdy et al.

Additionally, claim 18 recites an antioxidant extract obtained by a method comprising "extracting with an organic solvent selected from the group consisting of alcohols, esters, ketones and combinations thereof". At col. 1, lines 32-36, Purdy et al teach that "to extract these materials from sesame oil using solvents such as methyl alcohol...such solvents are not fully effective since they also dissolve certain materials from the sesame oil...". In this way, Purdy et al clearly teach away from an extract obtained by a method comprising "extracting with...alcohol..." as taught and recited in claim 18. Thus, Purdy et al neither disclose an extract possessing "sesamol 10-16%" nor do they disclose an extract obtained by "extracting with...alcohols.." as recited in claim 18. Accordingly, claim 18 is not anticipated or obvious over Purdy et al.

#### JP 62-172086

JP 62-172086 does not disclose a composition "...consisting of 5-20% lignan containing sesamol 10-16%; sesamin 60-75%; sesamolin 5-8.8%..."", as recited in claims 1 and 18, nor would the compound of JP 62-172086 inherently contain the recited ingredients of claims 1 and 18.

The presently claimed product would not be obtained in view of JP 62-172086 because this reference discloses a sesame extract for use as an antioxidant, wherein the antioxidant compounds of the extract are **Formula A** (tetrahydro- 1- (3- methoxy- 4- hydroxyphenyl)- 4- (3- methoxy- 4- hydroxyphenyl)- 4- (3,4- (methylenedioxy)-phenyl)- 1H,3H- furo-(3,4,- C) furan) and **Formula B** (tetrahydro- 1- (3- methoxy- 4- hydroxy- phenyl)- 4- (3,4- (methylenedioxy)phenyl)1H, 3H- furo(3,4,- C) furan), and neither Formula A nor Formula B is sesamol, sesamin or sesamolin as recited in claim 1.

The Applicants have enclosed a full translation of JP 62-172086. Given that the protocol of JP 62-172086 teaches adding an inorganic acid such as hydrochloric or sulfuric acid (see paragraph [015] of the enclosed translation), a step that is missing from the presently claimed invention, there is no reasonable expectation that the extract product of JP-62-172086 would inherently contain the same ingredients recited in claims 1 and 18, let alone in the claimed amounts. It would be immediately apparent to one skilled in the art that a product resulting from the present invention possessing the claimed components could not be presumed to also be in a product obtained from a method further including such a strong acid treatment as per JP 62-172086.

In view of the above, the Applicants respectfully submit that claims 1 and 18 are not anticipated by either Purdy et al or JP 62-172086. Accordingly, at least by virtue of their dependency on claim 1, claims 2-5 are not anticipated by Purdy et al or JP 62-172086.

**V. 35 USC 103(a)**

Claims 1-5 and 18 stand rejected under 35 USC 103(a) as being unpatentable over JP 62-172086. The Examiner states that if it is shown that the extract of JP 62-172086 would not be expected to possess the particular lignan profile and other components as called for in the instant claims, that it would have been well within the purview of a skilled artisan to have determined the particular lignan content and profile therein and that it follows that one skilled in the art at the time of the invention would have found it obvious to have arrived at the particular degree of antioxidant attributes achieved through the instant invention through routine experimental optimization. The Applicants respectfully disagree.

As stated above, JP 62-172086 teaches adding an inorganic acid such as hydrochloric or sulfuric acid after the organic solvent extraction step, and this acid step is not disclosed in the present application. Thus a full lignan analysis of the product of JP 62-172086 is not expected to contain the claimed lignans. Furthermore, with the lignan profile in hand, further optimization by one skilled in the art would not render the product of JP 62-172086 because after the strong acid treatment of the JP 62-172086, the claimed product cannot be easily derived. As disclosed in JP 62-172086, the antioxidant activity of the product is attributed to compounds of Formula A and Formula B and cannot be derived from the claimed lignan components as recited in claims 1 and 18.

Furthermore, JP 62-172086 does not provide motivation to "optimize" the claimed amounts because there is nothing in this reference or any reference that teaches that the claimed amounts are "optimum." Thus, the skilled person would not seek to arrive at the particular degree of antioxidant attribute achieved through the instant invention, when there is nothing in any of the cited references to teach him that these particular amounts are desirable?

Accordingly, the Applicants argue that claims 1 and 18 are patentable over JP 62-172086, and at least by virtue of their dependency on claim 1, claims 2-5 are also patentable over JP 62-172086. The Applicants request withdrawal of the rejection of these claims under 103(a) in view of JP 62-172086.

**VI. Conclusion**

The Applicants have complied with each and every requirement in the pending Office Action, and request allowance of the pending claims. All amendments and remarks herein are made without prejudice.

\* \* \* \* \*

The Commissioner is authorized to charge any additional fees which may be required or credit overpayment to deposit account no. 12-0415. In particular, if this response is not timely filed, then the Commissioner is authorized to treat this response as including a petition to extend the time period pursuant to 37 CFR 1.136 (a) requesting an extension of time of the number of months necessary to make this response timely filed and the petition fee due in connection therewith may be charged to deposit account no. 12-0415.

I hereby certify that this paper (and any enclosure referred to in this paper) is being transmitted electronically to the United States Patent and Trademark Office on

Respectfully submitted,

February 28, 2008

(Date of Transmission)

Stacey Dawson

(Name of Person Transmitting)

/Stacey Dawson/

(Signature)

February 28, 2008

(Date)

/Nicole Ballew Chang, 61,611/

Nicole Ballew Chang

Agent for Applicants

Reg. No. 61,611

LADAS & PARRY

5670 Wilshire Boulevard

Suite 2100

Los Angeles, CA 90036

(323) 934-2300

Enclosures: Full Translation of JP 62-172086  
Petition for 1-mo ext. under 37 CFR 1.136(a)

# TRANSLATION FROM JAPANESE

(19) Japanese Patent Office (JP)

(12) Published Laid-Open Patent Application (A)

(11) Published Patent Application Number  
S62-172086

(43) Date of Publication of Laid-Open Patent: July 29, 1987

(51) International Patent Cl. <sup>4</sup>	Classification Symbols	Internal Office Reg. Nos.
C 09 K 15/08		2115-4H
// C 11 B 5/00		7215-4H

Request for Examination, Not Requested

Number of Inventions: 2

(Total of 4 Pages [in the original])

(54) Title of the Invention  
Antioxidant

(21) Application Number: S61-13420

(22) Filing Date of Application: January 23, 1986

(72) Inventor:	Takeo Murui 5-13-7, Nishi Kamata Ota-ku, Tokyo
(72) Inventor:	Atsushi Inode 6-27-9, Mori Isogo-ku, Yokohama
(71) Filer of Application:	Nisshin Oil Mills, Ltd. 23-1, Shinkawa 1-chome Chuo-ku, Tokyo

## Specification

- [001] 1. Title of the Invention  
Antioxidant
- [002] 2. Claims  
(1) An antioxidant that has an oil soluble substance that is produced by treating sesame oil cake with an acid as an effective component.
- [003] (2) The antioxidant cited in Claim 1 characterized in that a fraction in which the sesame oil cake has been extracted with water or (and) an organic solvent is acid treated.
- [004] 3. Detailed Description of the Invention  
(a) Field of Industrial Utilization  
The present invention is one that relates to an antioxidant with which the oxidation of fats and oils is effectively suppressed.
- [005] (b) Prior Art  
It is well known that the fats and oils used for food and the processed oleaginous food products that contain large amounts of fats and oils used for food are oxidized by the oxygen in the air during the storage and distribution processes and this is a cause of a degradation of their flavors and a deterioration of their foodstuff properties. For many fats and oils as well as processed oleaginous food products, all kinds of antioxidants are frequently employed as food additives.
- [006] On the other hand, with regard to food additives, these are significant items for which societal concern has been high of late and the development of highly safe antioxidants has become a strong desire on the part of the consumers and manufacturers of oleaginous products.
- [007] (c) Problems of Prior Art To Be Solved by the Invention  
However, it cannot be said that the antioxidants that have been known up to this point have necessarily been satisfactory from the standpoint of safety.
- [008] Accordingly, the present invention has as its object the provision of an antioxidant that is highly safe and, moreover, possesses a capability to prevent oxidation that should be satisfactory.
- [009] (d) Means To Solve the Problems of Prior Art  
With the present invention, as a result of repeated diligent research in order to achieve the relevant object, the fact was discovered that an effective oxidation suppression action is shown by the oil soluble component that is obtained by the action of an inorganic acid on the sesame oil cake that is created when sesame oil is produced.

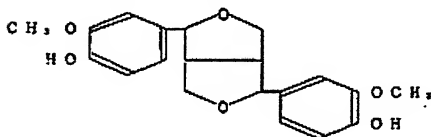
- [010] The present invention is one that was accomplished based on this kind of knowledge and is an antioxidant that has an oleaginous substance that is produced by the acid treatment of sesame oil cake as the effective component.
- [011] For the base material, sesame oil cakes, there are those that have been roasted under various conditions and those that are not roasted. In the present invention, any of these can be used as the base material but because with the roasted ones, there is thermal denaturation of the proteins, the solubility with respect to water is low and this is advantageous in later operations.
- [012] For the sesame oil cake, it is preferable that one that is a powder that is 100 mesh or less be used.
- [013] In addition, in the present invention, it is also possible to use a fraction that is obtained by extraction from the sesame oil cake discussed before with water, an organic solvent, or a mixture of these as the base material. In this case, as an effective organic solvent, in addition to alcohols that have strong polarity such as ethanol, propanol, isopropanol, methanol, butanol, and the like, solvents that do not have as strong a polarity as the alcohols such as chloroform, ethyl acetate, acetone, tetrahydrofuran, and the like are suitable and in those cases where these are mixed with water a water content of 5 to 50% is more effective.
- [014] Furthermore, the use of solvents that are not mixed with water is also possible and, in this case, it is advantageous if the unneeded components can be removed in advance prior to the acid treatment by washing the extract with water.
- [015] To the sesame oil cake or to the fraction that has been extracted from this with water or an organic solvent as described above, 5 to 20 times the amount of water or a solvent such as ethanol, methanol, propanol, isopropanol, and the like, and an inorganic acid such as hydrochloric acid and the like are added and the acidity is maintained while heating is done. It is necessary that the acid concentration and the temperature be maintained in an optimum state in order to make the action of the acid effective and with a strong acid such as hydrochloric acid, sulfuric acid, and the like, a 0.1 normal solution to 5 normal solution is preferable, while with a weak acid such as phosphoric acid and the like, a 0.5 normal solution to 5 normal solution is preferable. In addition, for the treatment temperature and time, it is preferable that the acid concentration and the temperature be set such that the treatment is completed in 5 to 20 hours at a temperature of 30° C to 100° C in all cases in which any of the acids is used. If at this time, an organic solvent described above such as ethanol and the like is used as a substitute for the water, because the solvent power with respect to the effective component in the base material is great, the treatment time may be made short but in those cases where the treatment temperature exceeds the boiling point of the organic solvent and in those cases where the temperature comes close to this, it is necessary to carry out the treatment in a vessel that has been hermetically sealed.



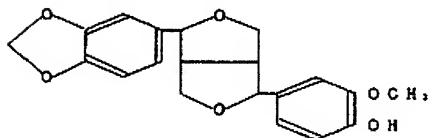
[016] After the treatment with an inorganic acid has been done, the residual acid in the treatment solution is neutralized using potassium hydroxide or sodium hydroxide and the insoluble residue is removed by means of centrifugal separation or filtering and the like. An inorganic solvent is used and the oil soluble component substances are extracted after the solution portion has been concentrated 5 to 10 times or with the solution portion as it is unconcentrated. As the organic solvent, ethyl acetate, chloroform, ethanol, propanol, isopropanol, butanol, and the like are suitable but in those cases where a water system has been used in the acid treatment, water insoluble ethyl acetate, chloroform, and the like are preferable. In those cases where an alcohol has been used in the acid treatment, the majority of the alcohol is distilled and removed and the solid substance that is obtained is extracted with any of the solvents described above after the insoluble residue has been eliminated.

[017] With regard to the effective component of the antioxidant in the present invention, the inventors ascertained the two types of compounds that are shown in the drawings below.

[018] A is tetrahydro-1-(3-methoxy-4-hydroxyphenyl)-4-(3-methoxy-4-hydroxyphenyl)-4-(3,4-(methylenedioxy)phenyl)1H, 3H-furo(3,4,-C)furan, and B is tetrahydro-1-(3-methoxy-4-hydroxyphenyl)-4-(3,4-(methylenedioxy)phenyl)1H, 3H-furo(3,4,-C)furan.



A



B

[019] (e) Working Examples

[020] Working Example 1

After the oil was expressed from Chinese sesame seeds that had been roasted, 1 kg of the oil cake from which the fat had been removed with hexane (residual oil content, 1% or less), which was then made into a powder of less than 100 mesh, was disposed in a 20 L stainless steel reaction vat and 12 L of water and 0.7 L of concentrated hydrochloric acid were added. This was hermetically sealed and gently stirred for 15 hours while being heated at 60° C. After a solution of potassium hydroxide was added and the pH had been adjusted to 7 after the time had passed, the treated solution was placed in a centrifuge and 8.3 L of a water phase was obtained. The water phase was placed in an evaporator and concentrated at less than 80° C to about 1 L. The concentrated solution was moved to a separating funnel and was extracted three times using 200 mL of ethyl acetate. The ethyl acetate phase was matched and drying was done with anhydrous sodium sulfate. The solvent was evaporated after concentration and 24.5 g of a dark brown solid substance was obtained.

[021] Working Example 2

One kg of sesame seed cake that had undergone the same processing as in Working Example 1 was disposed in a 10 L reaction vat. Five L of chloroform were added, a water cooling tube was attached, and circulation was done for one hour. The insoluble residue was removed after cooling by filtering and 4.1 L of extract solution were obtained. The solvent was evaporated from the extract liquid by means of an evaporator and 151 g of the extract were obtained. To the concentrate, 1 L of water and 25 ml of concentrated sulfuric acid were added and this was left to stand for 17 hours while being heated to a temperature of 40° C. Following neutralizing with potassium hydroxide after the time had passed, the same processing was done as in Working Example 1 and 28.0 g of a dark brown solid substance was obtained.

[022] Working Example 3

From 1 kg of sesame oil cake to which the same processing was done as in Working Example 2, 150 g of a chloroform soluble substance was obtained. Using a pressure resistant stainless steel reaction vessel, 1 L of ethanol and 50 mL of concentrated hydrochloric acid were added and this was hermetically sealed and heated at 100° C for six hours. The solution was placed in an evaporator following neutralization with a potassium hydroxide solution after the time had passed and the majority of the ethanol was distilled and removed. The residue was moved to a separation funnel using 300 mL ethyl acetate and after 100 mL of water were added and stirred, the ethyl acetate phase was segregated. After this, the same processing as in Working Example 1 was done and 25.5 g of a dark brown solid substance was obtained.

[023] Antioxidative Property Testing

[024] The antioxidative properties of the antioxidant that was obtained by means of Working Example 2 (hereafter, referred to as "this item") were investigated with regard to a linoleic acid hydroperoxidation reaction and compared with  $\alpha$ -tocopherol, which is a

representative natural antioxidant, and with BHA, which is a representative synthetic antioxidant.

- [025] The TBA method was used as the method for the evaluation of the antioxidative properties and the essential points are as follows.
- [026] In a 100 mL stopper equipped flask, 0.3 mL of linoleic acid (99%), 15 mL of ethanol, and 5 mL of water were disposed. Next, 1 mL of an ethanol solution of the antioxidant (5 mg/mL) was added and then 0.1 mL of t-butylhydroperoxide was added as a lipid hyperoxidation reaction induction factor. After maintaining the flask at 40° C for one hour, 2 mL of the solution were taken into a test tube. After 2 mL of a 0.67% aqueous solution of thiobarbital acid and 1 mL of 20% trifluoroacetic acid were added and mixed, this was held for 15 minutes in a boiling water bath. After cooling, this was placed in a centrifuge and the light absorption at 532 nm of the clear supernatant liquid was measured.
- [027] At the same time, an item with which the antioxidant was not added and the t-butylhydroperoxide was not used was made a blank and one to which the t-butylhydroperoxide was added was made the control. The percentage of hyperoxidation reaction inhibition by the antioxidant was derived using the following formula in which the light absorption of the blank and the control have been made B and C and the light absorption of the clear supernatant liquid when the antioxidant was added has been made A.

$$\text{In (\%)} = (B - A)/(B - C) \times 100$$

#### [028] Results

- [029] An evaluation was made using the method described above and the antioxidative properties of this item, the BHA, and the  $\alpha$ -tocopherol are shown in Table 1. The antioxidative property of this item is 87% and an antioxidative property that is equal to that of the  $\alpha$ -tocopherol and close to that of BHA was shown. It was ascertained that this item is an antioxidant of the top caliber.

Table 1

	Light Absorption	Antioxidative Property (%)
Blank	0.021	—
Control	0.691	0
This item	0.109	87
$\alpha$ -tocopherol	0.119	85
BHA	0.080	91

- [030] (f) Advantageous Result of the Invention  
Because the antioxidant of the present invention is an item that uses sesame oil cake,

which is a natural substance, as the base material, there are no problems from the standpoint of safety and, moreover, from the standpoint of practical use, a satisfactory oxidation prevention capability is shown.

Filer of Application: Nisshin Oil Mills, Ltd.

Amendment (Voluntary) Submitted March 12, 1986  
March 11, 1986

To: Commissioner of the Patent Office, Michiro Uga

1. Case Identifier

1986 Patent Application Number 13420

2. Title of the Invention

Antioxidant

3. Entity Making the Amendment

Relationship to the case: Filer of the Patent Application

Name: Nisshin Oil Mills, Ltd.

Address: 23-1, Shinkawa 1-chome, Chuo-ku, Tokyo

Representative: Mitsuo Fukawa [seal]

Incidentally, contact regarding this case should be made to the address listed below.

Name: Nisshin Oil Mills, Ltd., Laboratory

Postal Code: 221

Address: 1-3, Chiwaka-cho, Kanagawa-ku, Yokohama, Kanagawa Pref.

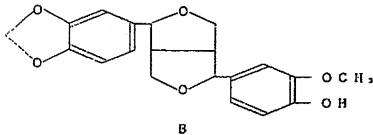
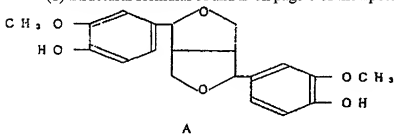
Telephone: 045 (461) 0181

4. Subject of the Amendment

(1) Detailed Description of the Invention field of the Specification

5. Details of the Amendment

(1) Structural formulas A and B on page 6 of the Specification are amended as follows.



(2) On page 9, line 9 of the Specification, "7% aqueous solution of thiobarbital acid" is amended to "7% aqueous solution of thiobarbituric acid."